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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT NO. 19
For Quarter Ending January 15, 1970

prepared by
R. W. Harrison

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-6474
R. L. Davies and P. L. Stone, Project Managers
Materials Section

NUCLEAR SYSTEMS PROGRAMS
SPACE SYSTEMS
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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October 15, 1969 to January 15, 1970

February 4, 1970

CONTRACT NAS 3-6474

NASA Lewis Research Center
Cleveland, Ohio
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FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. R. L. Davies and P. L. Stone of NASA-Lewis Research Center are the NASA Technical Managers.

The program is being administered for the General Electric Company by E. E. Hoffman, and R. W. Harrison is acting as the Program Manager. Personnel making major contributions to the program during the current reporting period include:

T-111 Corrosion Loop Operation - A. Losekamp, T. Irwin
Partial Pressure Gas Analysis - Dr. T. Lyon
1900°F Lithium Loop - J. Smith, J. Holowach, T. Irwin, J. Sharkey
Advanced Tantalum Alloy Capsule Tests - G. Brandenburg

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from October 15, 1969 to January 15, 1970. The primary task of this program is to fabricate, operate for 10,000 hours and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation include the containment alloy, T-111 (Ta-8W-2Hf) and the turbine candidate materials Mo-TZC and Cb-132M which are located in the turbine simulator of the two-phase potassium circuit of the system. The loop design will be similar to the Cb-1Zr Rankine System Corrosion Test Loop; a two-phase, forced convection, potassium corrosion test loop which has been tested under Contract NAS 3-2547.[(1)] Lithium is being heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop is being accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

a. Boiling temperature, 2050°F
b. Superheat temperature, 2150°F
c. Condensing temperature, 1400°F.
d. Subcooling temperature, 1000°F
e. Mass flow rate, 40 lb/hr
f. Boiler exit vapor velocity, 50 ft/sec
g. Average heat flux in plug (0-18 inches), 240,000 Btu/hr ft^2
h. Average heat flux in boiler (0-250 inches), 23,000 Btu/hr ft^2

In addition to the primary program task cited above the program also includes capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Re-1Hf) in both potassium and lithium.

[(1)] Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 7, R66SD3016, General Electric Company, Cincinnati, Ohio, May 1, 1968.

Also included in the program is the fabrication, 7500-hour operation and evaluation of a 1900°F, high flow velocity, pumped lithium loop designed to evaluate the compatibility of T-111 clad fuel specimens, ASTAR 811 type alloys, T-111, and W-Re-Mo Alloy 256,* at conditions simulating a lunar Brayton Reactor system.

*W-25 a/o Re-30 a/o Mo (W-29 w/o Re-18 w/o Mo)

II. SUMMARY

On January 15, 1970, the T-111 Rankine System Corrosion Test Loop successfully completed 8500 hours of operation. The loop has operated on automatic control for the past 2500 hours.

Fabrication and instrumentation of the 1900°F Lithium Loop was completed. The loop was placed in the vacuum chamber, pumped down, bake-out initiated and filled with lithium. Circulation of lithium was initiated on 1-15-70. A low power check out of the entire system has been started.

Testing of the refluxing potassium capsule tests of ASTAR 811C alloy is continuing.

III. PROGRAM STATUS

A. T-111 RANKINE SYSTEM CORROSION TEST LOOP

1. Loop Operating Temperature

On January 15, 1970 the T-111 Corrosion Test Loop successfully completed 8500 hours of the planned 10,000 hours of operation. The loop temperatures recorded at the 8500 hour test time are shown in Figure 1, and the temperatures of major interest are shown on the loop schematic in Figure 2. The performance of the loop has been excellent and power input to the lithium heater remained on automatic control for the past 2500 hours with excellent temperature control being maintained. A comparison of the operating conditions at 1000 hours, 5000 hours, and 7500 hours shown in Table I, further exemplifies the stability of the loop's performance.

2. Turbine Simulator Performance

The calculated vapor velocities of the turbine simulator nozzles at 7500 hours are presented in Table II for a mass flow rate of 36.7 lb/hr of potassium. The vapor velocity in the superheated first stage was 1090 ft/sec. The vapor velocity in the 88-percent-quality region ranged from a high of 1260 ft/sec in the second-stage nozzle to a low of 1119 ft/sec in the tenth stage nozzle. All vapor velocities were higher than the 1000 ft/sec design velocity.

The higher than design velocity is attributed to the lower than predicted vapor pressure at the inlet to the turbine simulator due to a higher than predicted pressure drop in the boiler. The higher than predicted pressure loss in the boiler is due to the high heat transfer rate in the 18-inch-long plug and the resulting high vapor quality in the entrance section of the boiler. For a given mass flow rate, the pressure drop in the tube is inversely proportional to the vapor density.

3. Test Chamber Environment-Partial Pressure Analysis

The chamber pressure and partial pressures of the various gaseous species in the test chamber during the period from 6000 to 8000 hours

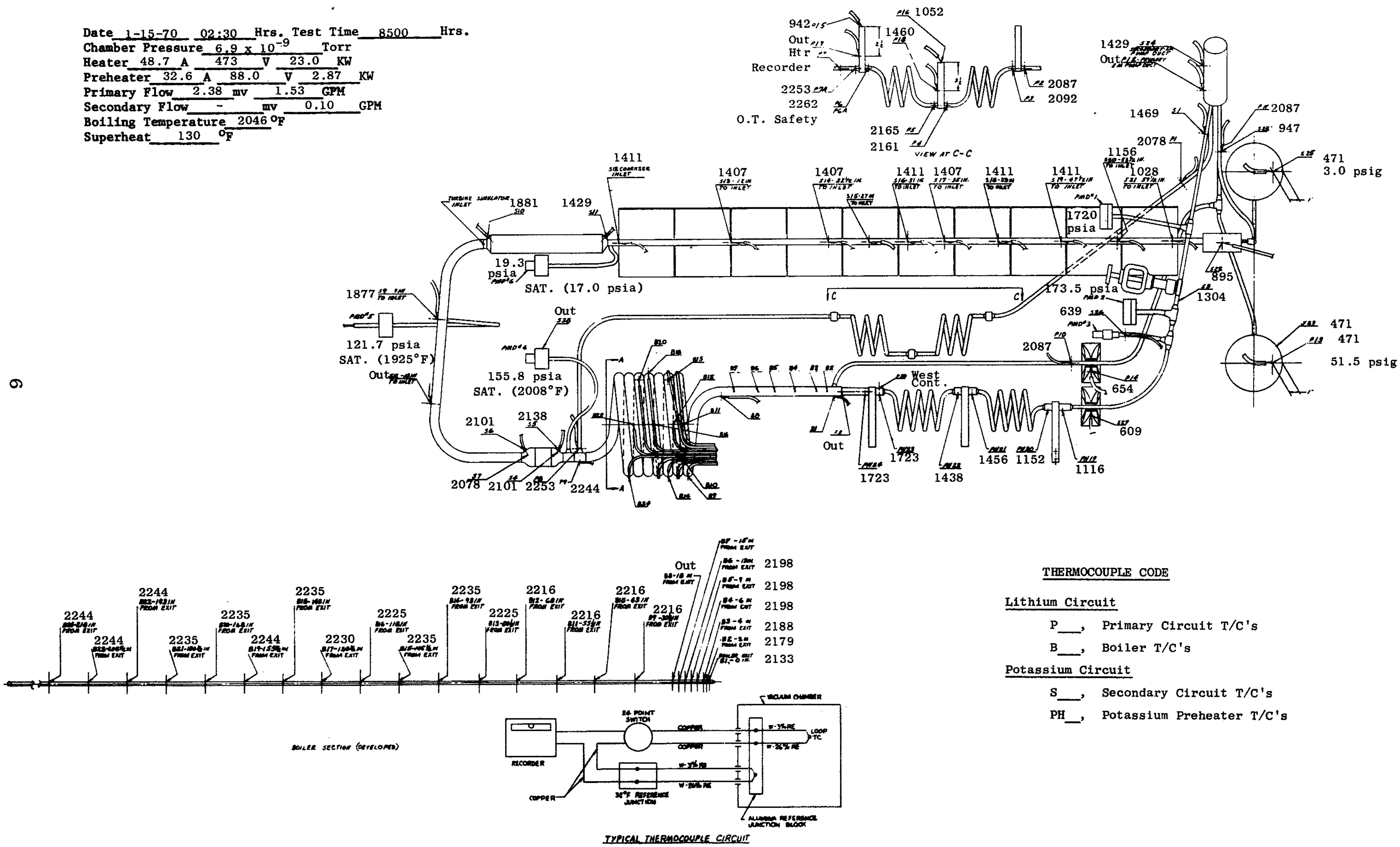

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Figure 1. T-111 Rankine System Corrosion Test Loop Thermocouple Instrumentation Layout.

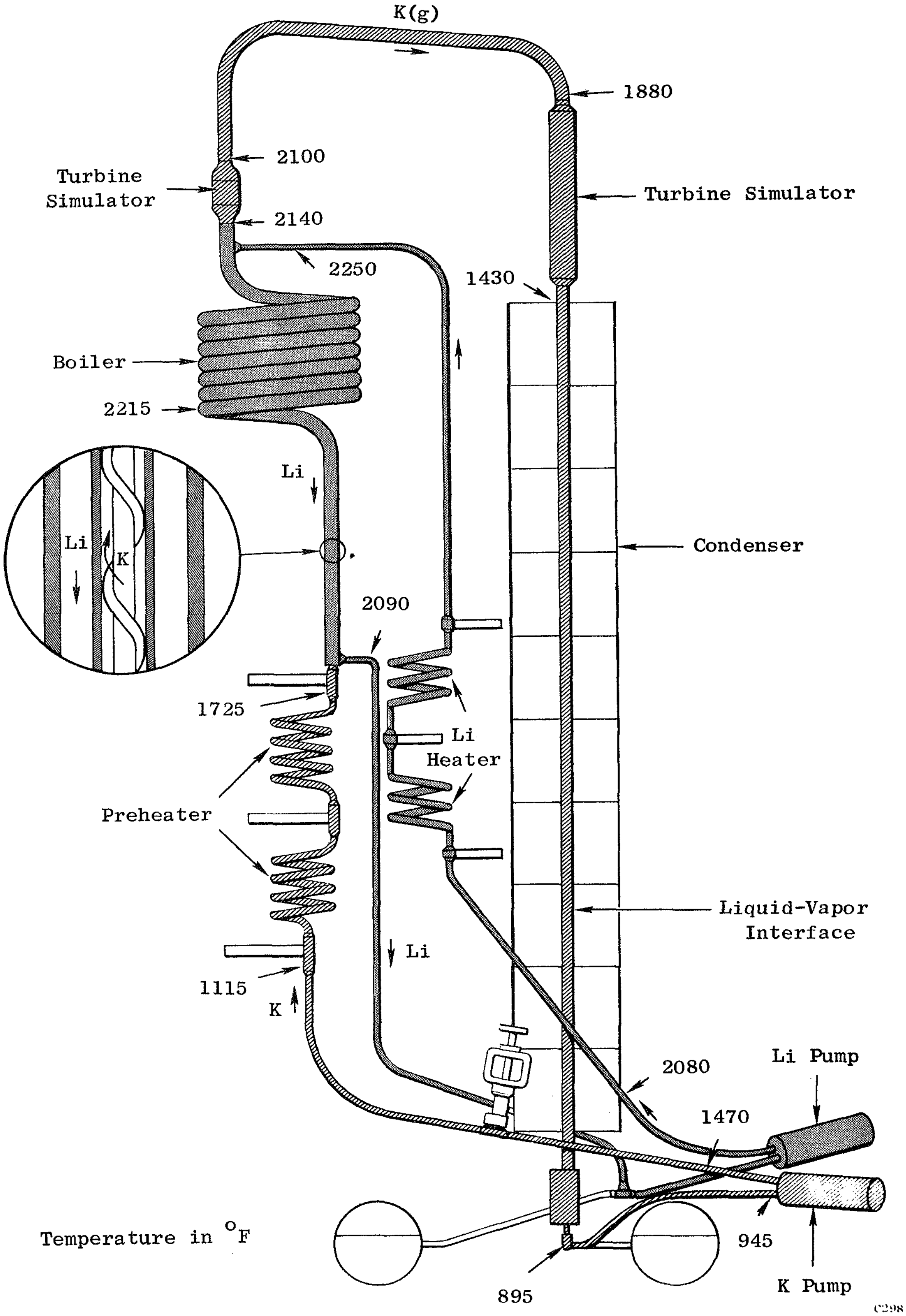

Figure 2. T-111 Corrosion Test Loop Operating Temperatures - 8500 Hours.

TABLE I

T-111 RANKINE SYSTEM CORROSION TEST LOOP PERFORMANCE

Date	3-8-69	8-22-69	12-4-69
Test Hours	1000	5000	7500
Lithium Flow Rate	205 lbs/hr	229 lbs/hr	242 lbs/hr
Lithium Temperature, In	2253 °F	2239 °F	2244 °F
Lithium Temperature, Out	2078 °F	2078 °F	2087 °F
Lithium ΔT	175 °F	161 °F	157 °F
Potassium Flow Rate	36 lbs/hr	38 lbs/hr	37 lbs/hr
Plug Boiling Temperature	2048 °F	2052 °F	2045 °F
Boiler Exit Vapor Temp.	2147 °F	2137 °F	2142 °F
Boiler Exit Saturation Temp.	2012 °F	2012 °F	2007 °F
Potassium Vapor Superheat	135 °F	125 °F	135 °F
Condensing Temperature	1416 °F	1411 °F	1411 °F
Potassium Heat Input			
1. Preheat	2280 Btu/hr	2410 Btu/hr	2430 Btu/hr
2. Heat of Vaporization	26,300 Btu/hr	27,600 Btu/hr	26,900 Btu/hr
3. Superheat	1040 Btu/hr	960 Btu/hr	990 Btu/hr
TOTAL	29,620 Btu/hr	30,970 Btu/hr	30,230 Btu/hr
Total Power to Lithium Heater	13.2 kw	13.8 kw	13.6 kw
Total Power to Potassium	8.7 kw	9.0 kw	8.9 kw
Net Heat Loss	4.5 kw	4.8 kw	4.7 kw

TABLE II

T-111 RANKINE SYSTEM CORROSION TEST LOOP TURBINE SIMULATOR PERFORMANCE AT 7500 HOURS

Nozzle Number	Material	Nozzle Diameter (R.T.), Inch	Inlet Temperature °F	Exit Pressure psia	Vapor Velocity ft/sec
1	Mo-TZC	0.0892	2142	110.0	1090
2	Mo-TZC	0.0881	1890	91.0	1260
3	Mo-TZC	0.0964	1834	74.0	1270
4	Mo-TZC	0.1083	1774	60.0	1216
5	Mo-TZC	0.1181	1716	49.5	1227
6	Cb-132M	0.1292	1664	40.0	1245
7	Mo-TZC	0.1457	1611	32.5	1184
8	Mo-TZC	0.1598	1562	26.3	1198
9	Cb-132M	0.1784	1514	21.5	1158
10	Mo-TZC	0.1986	1471	17.6	1119

of loop operation are shown in Figure 3. Similar data for the period from 0 to 6000 hours of loop operation were given in the preceding quarterly reports.[1,2,3] In these Figures, data points are plotted at 250 hour intervals for the sake of clarity, even though residual gas analyses are obtained every eight hours during loop operation. As shown in Figure 3, the total pressure has been gradually decreasing over the entire test period up to about 6750 hours as a result of the diminishing hydrogen partial pressure. At about 6750 hours the hydrogen partial pressure, which continues to be the predominant gas in the system, essentially leveled off as did the total pressure. The pressure has remained essentially constant for the period 6750 through 8000 hours.

4. <u>Loop Operation</u>

The loop continues to operate stably and trouble free, and has been operating on automatic control for the past 2500 hours.

A malfunction in the Simplytrol on the primary EM pump windings caused the loop to shutdown at 0515 on 1-10-70; the loop was brought back to test conditions with a loss of approximately one hour of test time. During the shutdown the secondary flowmeter became inoperative; however, this not a serious problem since the flow can be calculated from ΔT measurements in the loop. Approval to operate under these conditions has been obtained from the NASA program managers.

---

(1) <u>Advanced Refractory Alloy Corrosion Loop Program</u>, Quarterly Progress Report No. 16 for Period Ending April 15, 1969, NASA Contract NAS 3-6474, NASA-CR-72560 (GESP-258).

(2) <u>Advanced Refractory Alloy Corrosion Loop Program</u>, Quarterly Progress Report No. 17 for Period Ending July 15, 1969, NASA Contract NAS 3-6474, NASA-CR-72592 (GESP-303).

(3) <u>Advanced Refractory Alloy Corrosion Loop Program</u>, Quarterly Progress Report No. 18 for Period Ending October 15, 1969, NASA Contract NAS 3-6474, NASA-CR-72620 (GESP-376).

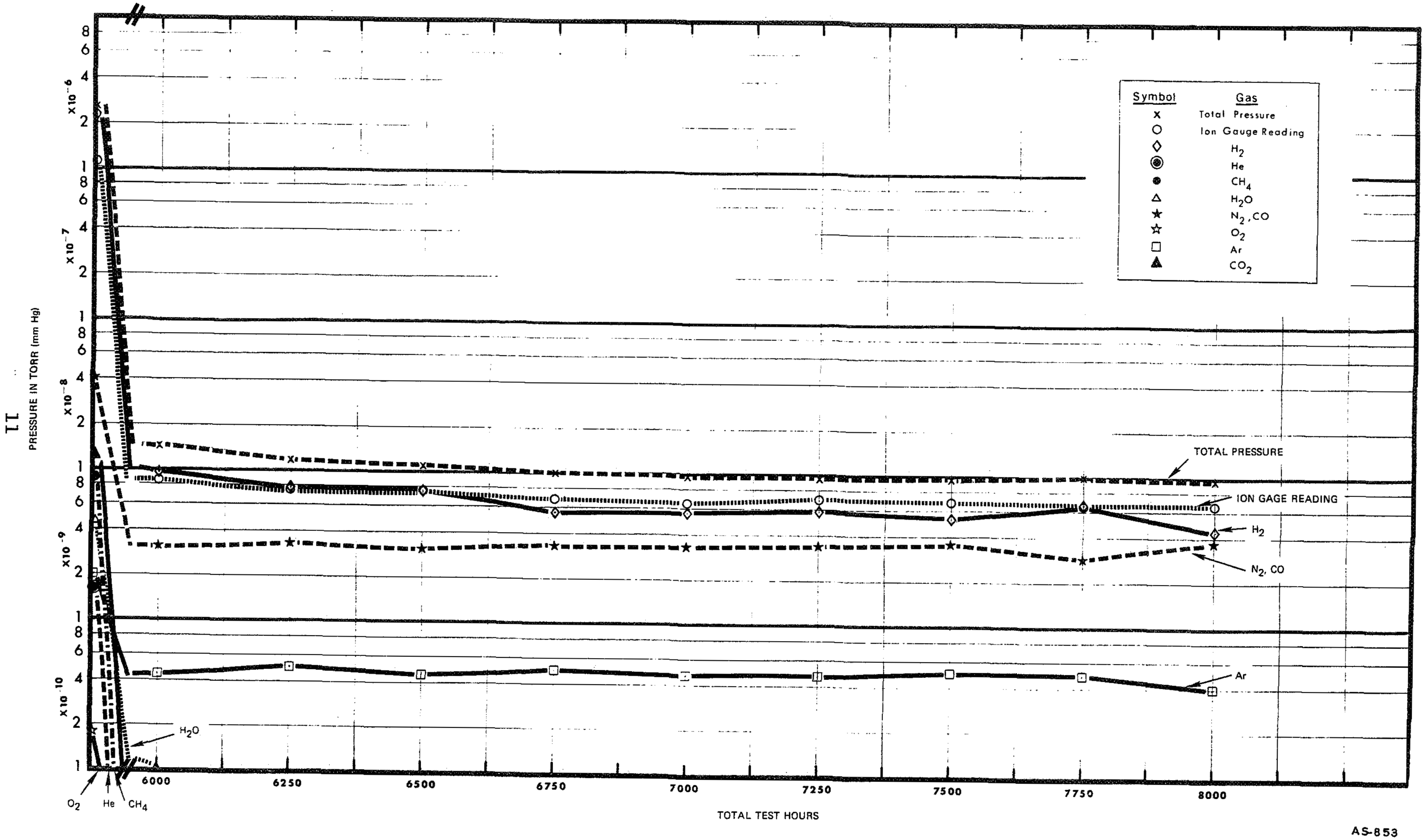

Figure 3. Test Chamber Environment During Testing of the T-111 Rankine System Corrosion Test Loop.

B. 1900°F LITHIUM LOOP

During the current reporting period, final assembly and instrumentation of the 1900°F Lithium Loop was completed. The loop was placed in the vacuum chamber, evacuated, baked-out, leak checked, and filled with lithium. Low power was applied to the heater and all safety circuits checked-out; calibration of the thermocouples and flowmeters was initiated.

The final fabrication, instrumentation, filling, pump-down, and check-out procedures are described in detail below.

1. Final Fabrication

Following heat treatment of the assembled loop (except EM pump and surge tank) for 1 hour at 2400°F in the vacuum chamber at Union Carbide Corp.,* the assembly was returned to Evendale and bolted into the stainless steel support structure. The EM pump and surge tank were fit into their proper locations, held in place with temporary supports, and transferred to the welding chamber. All final refractory metal weld and postweld anneals were then performed according to GE-NSP Specification 03-0025-00-A. These welds were subsequently radiographed and helium mass spectrometer leak checked and found to be free of defects. The loop and support structure were then mounted on the 24-inch diameter spool piece, and transferred to the portable laminar flow clean room facility as shown in Figure 4. A close-up view of the assembled loop is shown in Figure 5 prior to application of thermal insulation and instrumentation.

2. Thermal Insulation and Instrumentation

Thermal insulation consisting of multiple layers of Cb-1Zr foil was simultaneously applied to the loop as the thermocouples were installed. The foil used on all circular pipe sections was 0.002 inch thick x 0.5 inch wide which had been dimpled by passing the foil between a hardened steel, coarse knurled roller working against a hard plastic sheet. The effective thickness of the foil after dimpling was between 0.009 to 0.012 inch. The insulation was attached by spot welding the foil to the loop and to itself as succeeding layers were applied. A minimum number of spot welds were used to minimize conduction heat losses through the

*Now Stellite Division of the Cabot Corporation, Kokomo, Indiana.

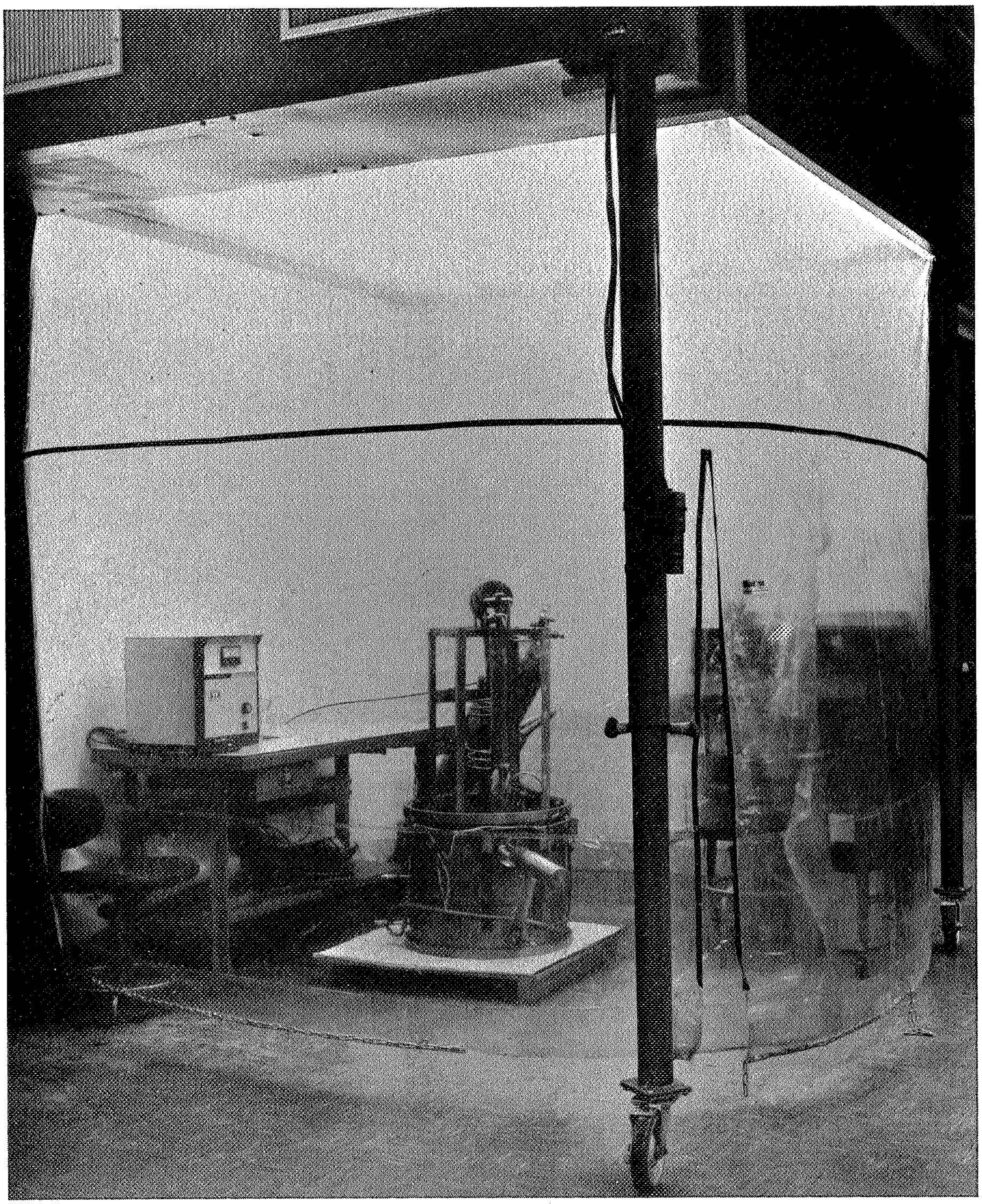

Figure 4. 1900°F Lithium Loop During Instrumentation and Insulation in the Clean Room Facility. (69-11-9A)

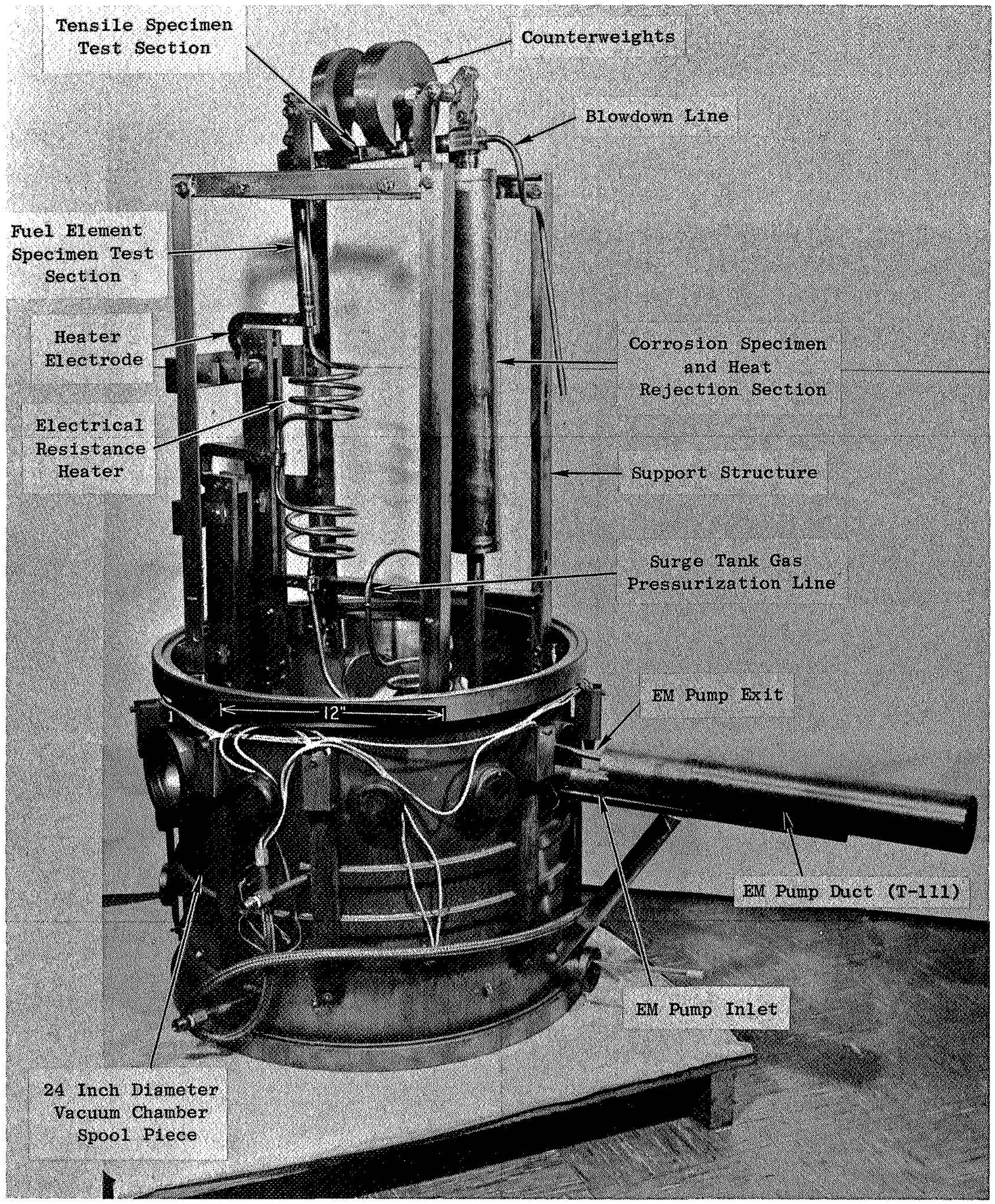

Figure 5. Assembled T-111 Alloy 1900°F Lithium Loop Ready for Instrumentation and Insulation. (69-11-9B)

foil. A molybdenum spot welder electrode was used to avoid contamination of the foil surfaces with copper and an argon cover gas was used to protect all welded areas from oxidation. The entire loop was insulated as described above except for the corrosion specimen section which is to act as the heat rejection section of the loop. The outer surface of the 3-inch diameter lithium containment tube which surrounds the tube containing the corrosion specimens was grit blasted with Al_2O_3 to produce an effective emittance of approximately 0.4.

All thermocouples installed on the loop were split junction W-25Re/W-3Re thermocouples which were insulated with Al_2O_3 and applied according to GE-NSP Specification 03-0019-00-A. The completely assembled, instrumented and insulated loop is shown in Figure 6. Details of the thermocouple feedthroughs and cold junction reference temperature compensation were described in the Test Plan.[4]

At the completion of this step, the loop was covered with a protective polyethylene bag and moved from the fabrication and assembly area of Building 700 to the test area in Building 314. The spool piece and loop were clamped to the 24-inch diameter vacuum chamber sump and all final electrical, water and mechanical connections were made. All thermocouples were then checked for continuity to the control panel. The complete loop and vacuum system ready for enclosure within the bell jar is shown in Figure 7.

The bell jar was located in place on the spool piece, clamped down, and pump down of the chamber was initiated. Vacuum chamber bake-outs and the surge tank heater were turned on when the chamber pressure reached 1×10^{-7} torr and bakeout was allowed to continue until after the loop was filled with lithium as described below. Prior to filling, the loop was pressurized to 50 psig and an argon mass scan taken in the vacuum chamber to verify that no leaks were present prior to exposure to lithium.

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(4) "Test Plan for 1900°F Lithium Loop, Advanced Refractory Alloy Corrosion Loop Program," NASA Contract NAS 3-6474 (GESP-394).

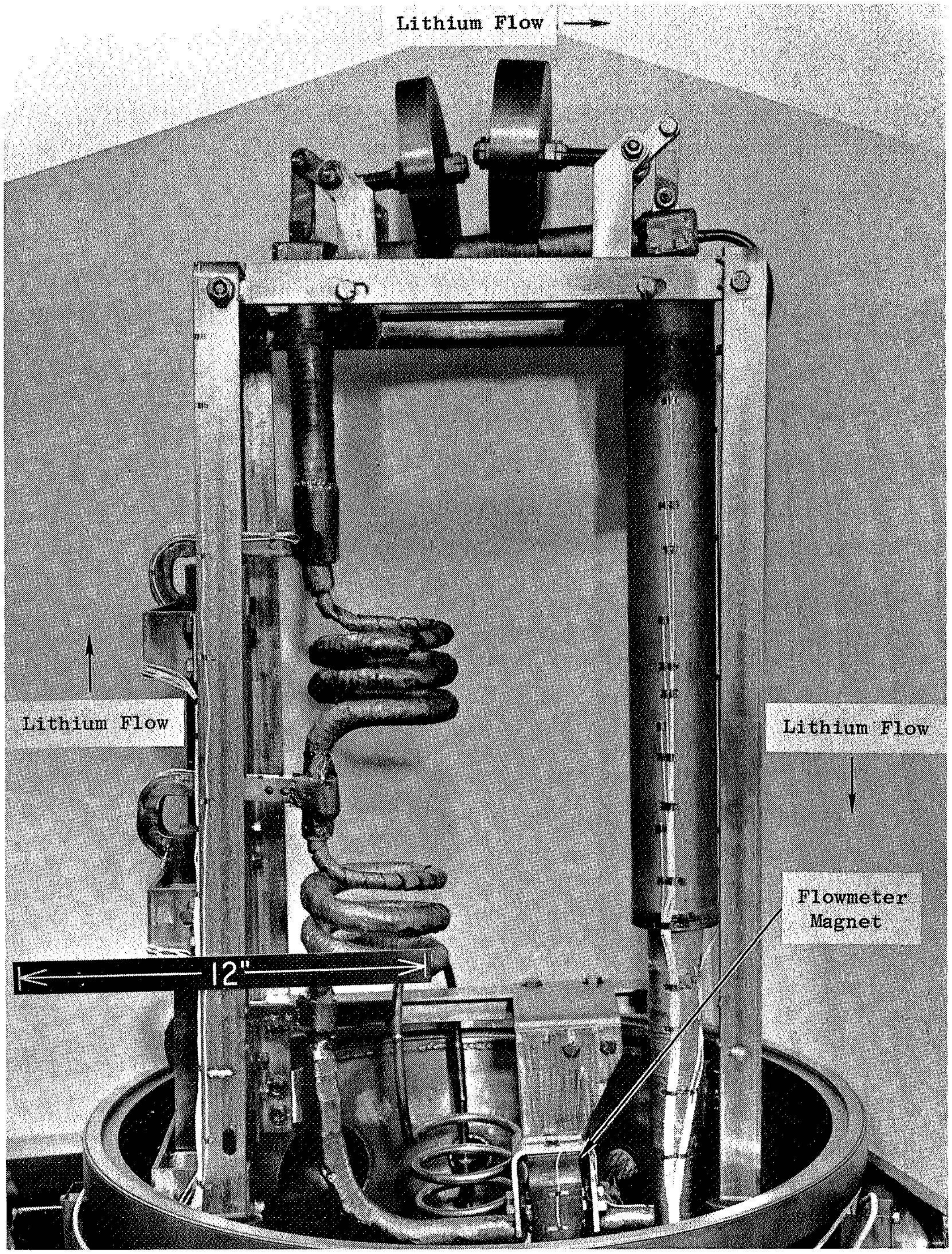

Figure 6. Completely Instrumented and Insulated 1900°F Lithium Loop. (70-1-2A)

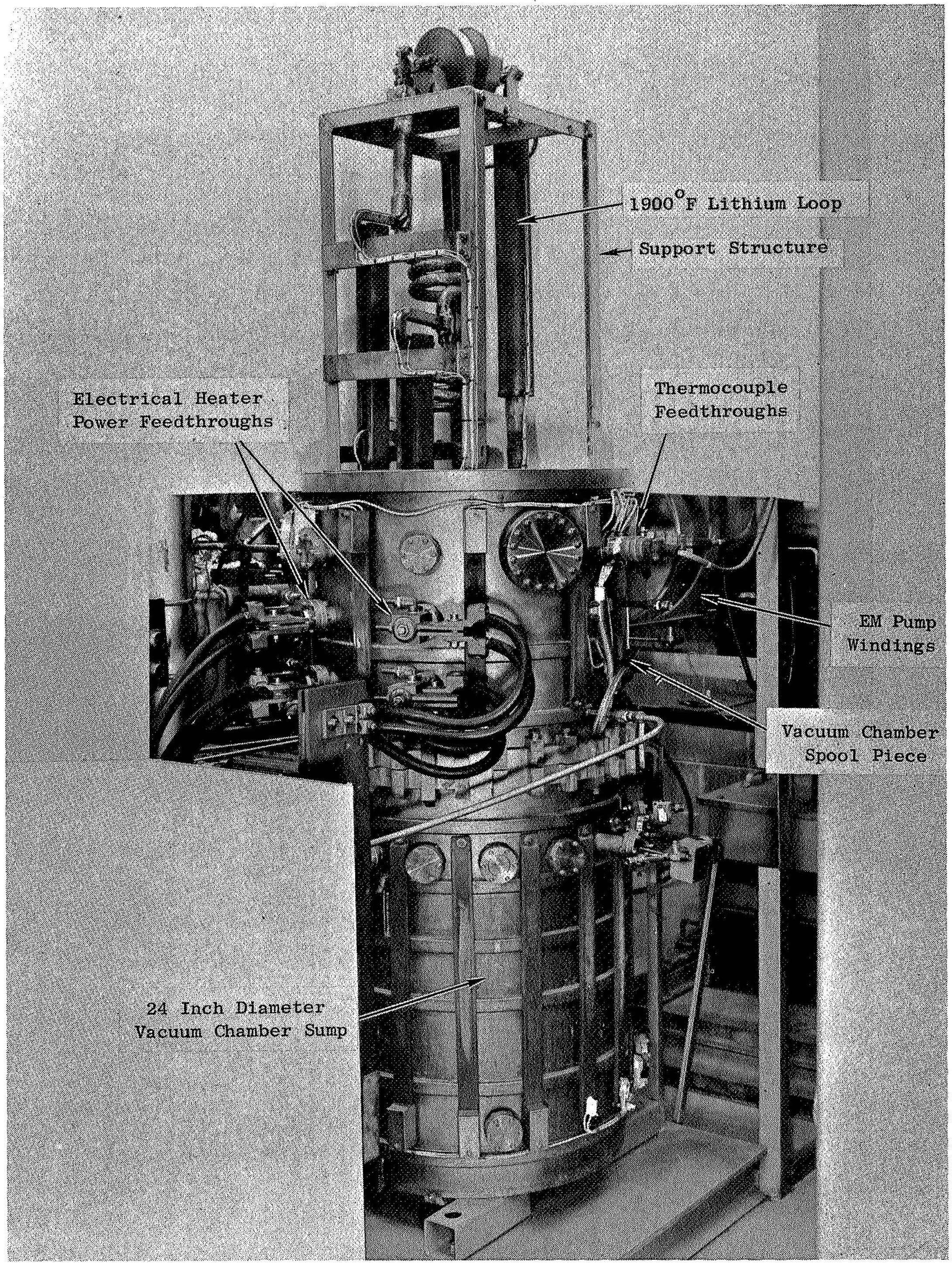

Figure 7. $1900^{\circ}F$ Lithium Loop Located on Vacuum Chamber Just Prior to Positioning of the Bell Jar. (70-1-2B)

3. Lithium Purification and Loop Filling

With the spool piece located in place on the vacuum chamber sump the various stainless steel gas and liquid metal lines were welded between the loop and the liquid metal transfer system. Bakeout and pump down of all loop, fill and transfer lines was begun immediately. The lithium used to fill the loop was high purity, vacuum distilled and hot trapped of an analysis shown in Table III.

Filling procedure was as described in detail in the Test Plan.[4] In general the procedure was as follows:

a) Heat loop with vacuum chamber bakeout and surge tank heater to at least 400°F.

b) Sample lithium from charge pot for oxygen and nitrogen.

c) Fill surge tank with approximately 3500 cc of lithium.

d) Pressurize surge tank to 50 psig thus filling the loop with lithium and allow to circulate 30 minutes.

e) Dump lithium into surge tank.

f) Repeat d and e for a total of three flushes.

g) Sample lithium from surge tank for oxygen and nitrogen analyses.

The analysis of the lithium sample taken from the loop after flushing indicated 28 ppm oxygen and 2-3 ppm nitrogen, both of which are well within the allowable maximum limits.

Filling was completed on January 14, 1970; verification of the acceptable chemical analysis was received on January 15, 1970 at which time a low power check out of all safety and control circuits was initiated. Loop temperature will be increased at a rate such that the chamber pressure does not exceed acceptable limits until the desired test temperature of 1900°F is reached.*

(4) "Test Plan for 1900°F Lithium Loop, Advanced Refractory Alloy Corrosion Loop Program," NASA Contract NAS 3-6474, GESP-394.

*The 1900°F Lithium Loop was brought to test conditions on January 31, 1970.

TABLE III

LITHIUM ANALYSIS - 1900°F LITHIUM LOOP

	Concentration, ppm		
	From Still Receiver[a]	From Transfer System[b]	From Loop[c]
Oxygen	32,49	25	28
Nitrogen	3,4	2,2	2,3
Carbon	88	86	76
Silver	< 5	< 5	< 5
Aluminum	25	5	5
Boron	< 75	< 75	< 75
Barium	< 75	< 75	< 50
Beryllium	< 5	< 5	< 5
Calcium	50	5	25
Columbium	< 25	< 25	< 25
Cobalt	< 5	< 5	< 5
Chromium	< 5	< 5	< 5
Copper	5	5	5
Iron	5	< 5	5
Magnesium	5	5	5
Manganese	< 5	< 5	< 5
Molybdenum	< 5	< 5	< 5
Sodium	< 125	< 125	--
Nickel	< 5	< 5	< 5
Lead	< 50	< 50	< 50
Silicon	5	5	< 5
Tin	< 25	< 25	< 25
Strontium	25	< 5	50
Titanium	< 25	< 25	< 25
Vanadium	< 25	< 25	< 25
Zirconium	< 25	< 25	< 25

(a) Sample No. 2767 - Lithium from still receiver prior to 1900°F loop fill.

(b) Sample No. 2776 - Lithium from 1900°F loop fill system flush at 500°F.

(c) Sample No. 2777 - Lithium from 1900°F loop flush at 450°F.

C. ADVANCED TANTALUM ALLOY CAPSULE TESTS

1. Potassium Reflux Capsules

Two ASTAR 811C potassium reflux capsules continue on test at 2200°F. On January 15, 1970, 4050 hours of testing were accumulated of the 5000 hour planned test. The chamber pressure at that time was 6.2×10^{-9} torr.

The condensing rate for each of the capsules is determined periodically by dividing the heat rejected per unit of condensing area by the heat of condensation for potassium at the capsule temperature. The heat rejected by the capsule to the water-cooled sink is calculated using the following equation:

$$Q_{12} = C_p M(\Delta T)$$

where:

C_p = Heat capacity of the cooling water (at the average water temperature)

M = Mass of cooling water per unit time

ΔT = Temperature difference of inlet and outlet cooling water.

The condensing rates and operating conditions for the two capsules are very similar as presented in Table IV.

Capsule testing will be completed on February 24, 1970 at which time the capsules will be removed from the test facility for posttest evaluation.

TABLE IV

CONDENSING RATES AND OPERATION CONDITIONS FOR ASTAR 811C POTASSIUM REFLUX CAPSULES

Capsule 1	
Average Condensing Temperature	2195°F
Condensing Rate at Average Temperature	31.0 ± 0.39 lbs/hr ft^{2}*
Average Power Input	1572 watts
Average Power Loss	60.0%
Vacuum	6.2×10^{-9} torr
Total Test Hours	4050 hours
Capsule 2	
Average Condensing Temperature	2175°F
Condensing Rate at Average Temperature	32.7 ± 0.36 lbs/hr ft^{2}*
Average Power Input	1516 watts
Average Power Loss	56.2%
Vacuum	6.2×10^{-9} torr
Total Test Hours	4050 hours

*Limits represent 95% confidence level based on least 5 squares linear analysis

FUTURE PLANS

A. Complete 10,000 hours operation of the T-111 Rankine System Corrosion Test Loop.

B. Complete final check-out and calibration of the 1900°F Lithium Loop and bring the loop to full operating conditions.

C. Complete 5000 hours of testing - ASTAR 811C Refluxing Potassium Capsules.

PUBLISHED REPORTS

Quarterly Progress	For Quarter Ending
Report No. 1 (NASA-CR-54477)	July 15, 1965
Report No. 2 (NASA-CR-54845)	October 15, 1965
Report No. 3 (NASA-CR-54911)	January 15, 1966
Report No. 4 (NASA-CR-72029)	April 15, 1966
Report No. 5 (NASA-CR-72057)	July 15, 1966
Report No. 6 (NASA-CR-72177)	October 15, 1966
Report No. 7 (NASA-CR-72230)	January 15, 1967
Report No. 8 (NASA-CR-72335)	April 15, 1967
Report No. 9 (NASA-CR-72336)	July 15, 1967
Report No. 10 (NASA-CR-72352)	October 15, 1967
Report No. 11 (NASA-CR-72383)	January 15, 1968
Report No. 12 (NASA-CR-72452)	April 15, 1968
Report No. 13 (NASA-CR-72483)	July 15, 1968
Report No. 14 (NASA-CR-72505)	October 15, 1968
Report No. 15 (NASA-CR-72527)	January 15, 1969
Report No. 16 (NASA-CR-72560)	April 15, 1969
Report No. 17 (NASA-CR-72592)	July 15, 1969
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